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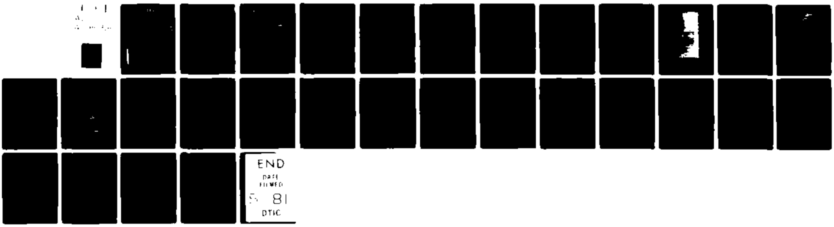

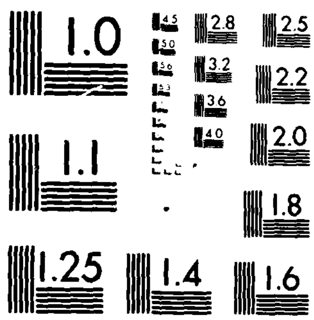
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INFRARED ANALYSIS OF GASOLINE/ALCOHOL BLENDS

INTERIM REPORT
AFLRL No. 134

by

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Under contract to

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An infrared method for qualitative and quantitative determination of alcohols in gasoline/alcohol blends has been developed. The method has high potential for speed, low cost, and specificity.

Gasoline containing methanol, ethanol, iso-propanol, t-butanol, methyl t-butyl ether, and methyl iso-butyl ketone (MIBK) can be analyzed using a computing infrared spectrophotometer.

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20. ABSTRACT (Cont'd)

Due to variable composition of different gasolines, each base gasoline analyzed required a new quantitative program to be established, using standards prepared with that particular gasoline.

FOREWORD

The work reported herein was conducted at the U.S. Army Fuels and Lubricants Research Laboratory (USAFLRL) located at Southwest Research Institute, San Antonio, Texas under Contract No. DAAK70-80-C-0001. The contracting officer's representative was Mr. F.W. Schaekel, Energy and Water Resources Laboratory, USAMERADCOM, DRDME-GL, Fort Belvoir, Virginia.

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I. INTRODUCTION

The use of alcohols as fuel extenders is being considered to reduce U.S. dependence on foreign oil and to prolong the existing supply of domestic petroleum. The use of ethanol in particular has been intensively researched for possible economic and environmental advantages over neat gasoline. For example, a blend of 10 vol% ethanol in gasoline increases the antiknock index 2-5 octane numbers over that of the base gasoline and reduces the hydrocarbon and carbon monoxide exhaust emissions. However, deleterious effects can arise from incompatibilities of ethanol with different gasolines and elastomers which may be accentuated in storage, routine handling and distribution. As a result, other oxygenates such as methanol, iso-propanol, t-butanol, methyl-t-butyl ether, and methyl-iso-butyl ketone (MIBK) blended with gasoline alone or in various combinations are being considered to determine any advantages over ethanol/gasoline blends.

Several analytical methods dealing with gasohol as well as synfuels and conventional fossil fuels have been studied. Various qualitative and quantitative methods for the determination of alcohols in gasohols are being evaluated. Infrared spectrophotometry has been found to be a useful method for the quantitative determination of the volume percent of oxygenates in a gasoline blend for specification conformation.

II. APPROACH

An infrared spectroscopic method for oxygenates has been emphasized because of its high potential for speed, low cost, and specificity. Figure 1 shows a typical IR spectrum of a fuel blend. Using the PEAKPICK MODE of a Beckman Microlab 620MX computing infrared spectrophotometer, analytical frequencies for each component were chosen where there was little or no interference from other components. Figure 2 shows an instrument printout, and Table 1 lists

TABLE 1. ALCOHOL ANALYTE BAND NUMBERS

Component	Analytical Frequency, cm^{-1}
Gasoline	967
Methanol	1030
Ethanol	882
iso-propanol	952
_t_-butanol	914
Methyl-_t_-butyl ether	1086

these chosen frequencies. Three background frequencies, 1255 cm^{-1}, 1230 cm^{-1}, and 860 cm^{-1}, were also selected which bracket the analytical frequencies and are used for baseline correction. Figure 3 shows the IR spectrum of a fuel blend with the analytical and baseline frequencies marked. Using these background points, the net percent transmittance at each frequency is obtained, converted to absorbance values, and normalized with respect to unit path length and concentration.

Each set of values was stored in array rows of a net normalized absorbance matrix and was later used to calculate the percentages of alcohols in unknown samples.

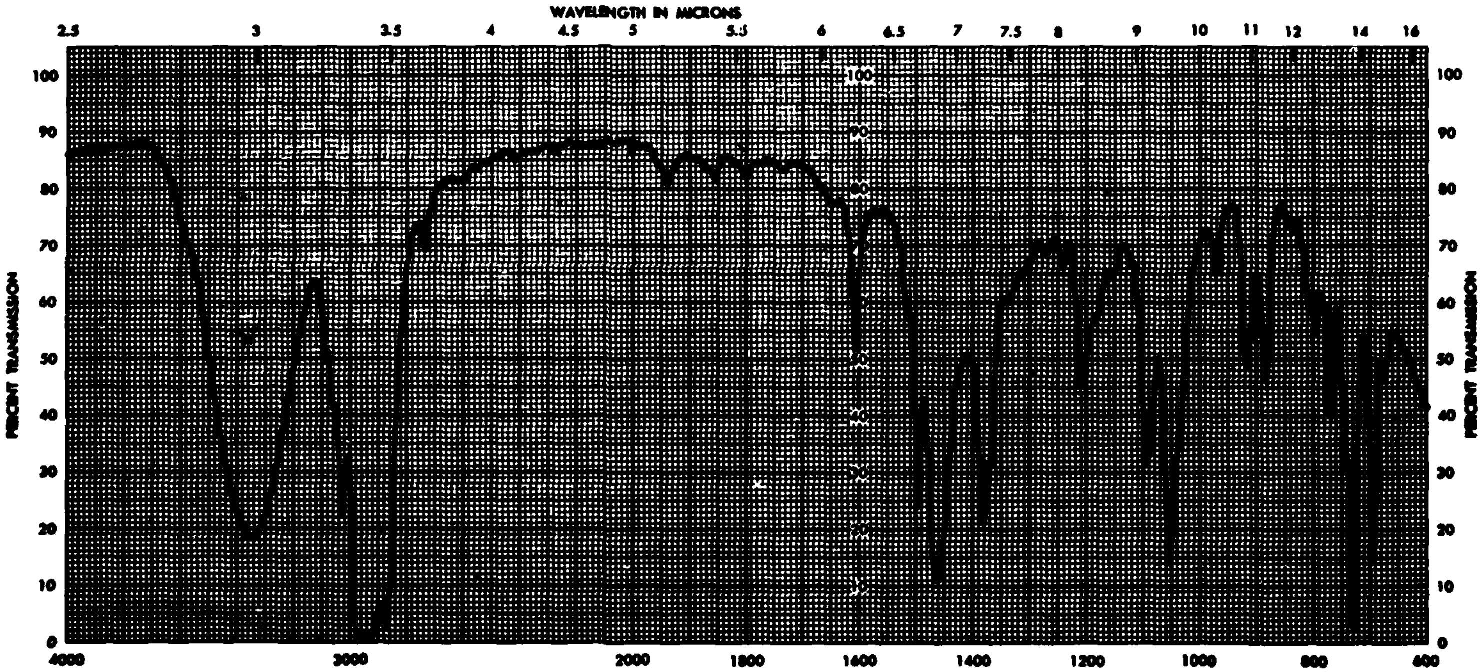

FIGURE 1. TYPICAL INFRARED SPECTRUM OF A FUEL BLEND

```
SCAN ROUTINES: 1-SCAN 2-REPSCAN
3-PEAKPICK 4-TIMEDRIVE

############  PEAKPICK   ############
SET %T DISCRIMINATOR
50
ENTER SCANNING PARAMETERS THEN
PUSH START

SCAN PARAMETERS: 1-RANGE 2-SPEED
3-CHART FORMAT 4-SLIT 5-GAIN
ENTER RANGE
2000 TO 0600
1604CM-1         46.7%T
1512CM-1         49.9%T
1494CM-1         22.2%T
1459CM-1          9.5%T
1378CM-1         26.0%T
1080CM-1         49.7%T
1030CM-1          6.2%T
0967CM-1         48.7%T
0888CM-1         50.0%T
0803CM-1         44.4%T
0793CM-1         37.7%T
0767CM-1         24.1%T
0727CM-1          1.1%T
0689CM-1          1.5%T
0667CM-1          0.2%T
0640CM-1          0.5%T
0633CM-1          0.4%T
0618CM-1          0.4%T
0612CM-1          0.5%T
0608CM-1          0.5%T
```

FIGURE 2. INSTRUMENT PRINTOUT OF PEAKPICK MODE

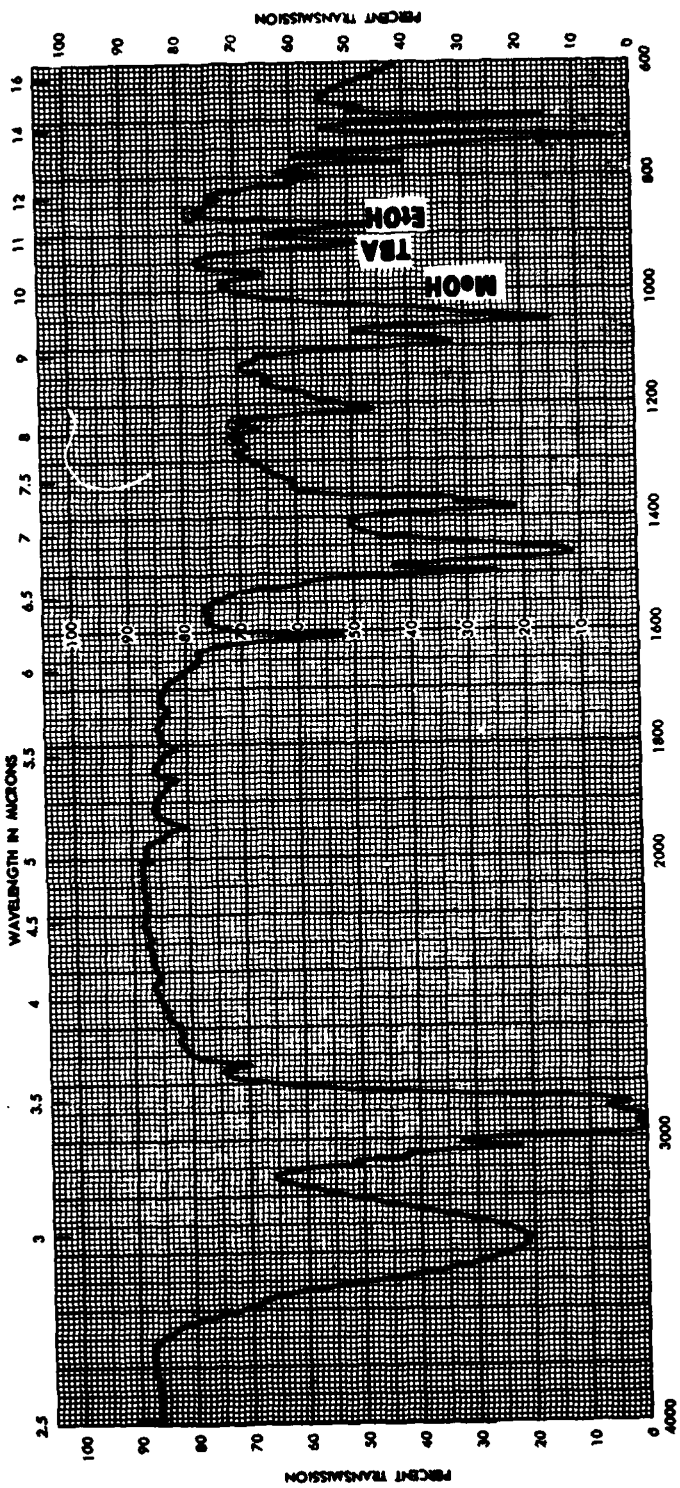

FIGURE 3. INFRARED SPECTRUM OF A FUEL BLEND

III. EXPERIMENTAL

All calculations were performed using a matrix set up for each gasoline by the Beckman Microlab 620MX computing infrared spectrophotometer. An unknown sample was analyzed using a precisely calibrated (0.025 cm nominal) sealed cell to prevent evaporation. The cell was filled completely, including the syringe fittings to ensure that absolutely no air was trapped.

A NaCl cell was used with no difficulty since no analytical frequencies were needed between 700 and 600 cm^{-1}. However, the attack on the cell by methanol was too severe and required the use of Irtran cells. After the analysis was completed, the cell was cleaned with heptane and dried.

A. Standards

To establish a procedure for preparing standards, spectra of varying concentrations of each alcohol in a gasoline were obtained, with Figure 4 showing a low and high standard for methanol. The net peak absorbance was plotted as a function of concentration for each alcohol, and a linear response with a non-zero intercept was obtained in each case. Figure 5 shows the plot for methanol. As a result, a low and high calibration standard is used for each component in a non-zero intercept method.

B. Instrumental

Using the quantitative routines on the computing infrared spectrophotometer, a POINT PROGRAM MODE and NORMALIZE MODE are run for each component. A POINT PROGRAM operation obtains transmittance measurement at prescribed wavelengths and stores these data for further use. A POINT PROGRAM printout is shown in Figure 6. The NORMALIZE MODE operation retrieves the data stored by the POINT PROGRAM, and with cell pathlength and component concentration values supplied, converts all data to a "normalized" standard format for future use. These normalized data take into account variations in cell pathlengths in subsequent analyses and the concentration of components in the standardization runs. Although used as the solvent for the alcohols, the gasoline was run as an undiluted component due to some absorbance at the analytical frequencies and

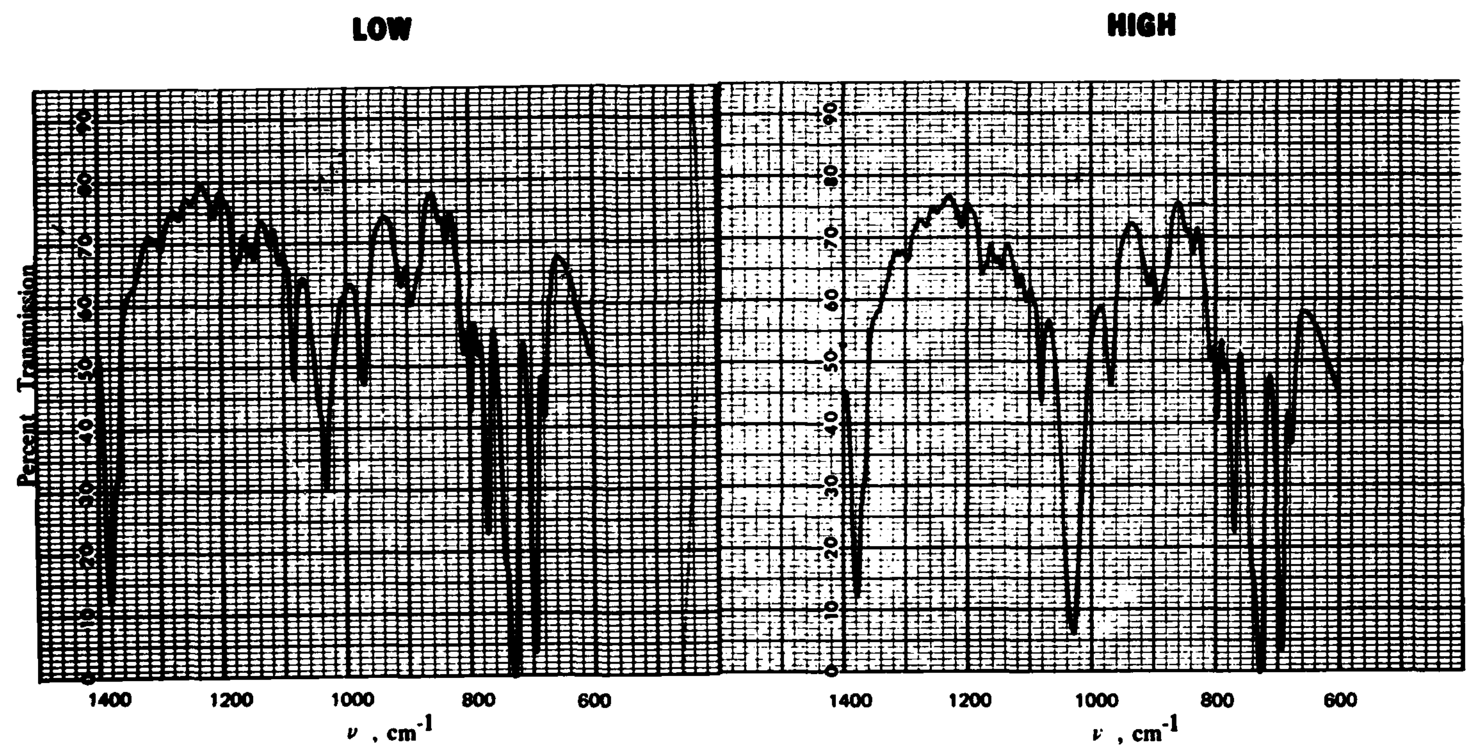

FIGURE 4. INFRARED SPECTRA OF LOW AND HIGH STANDARDS FOR METHANOL

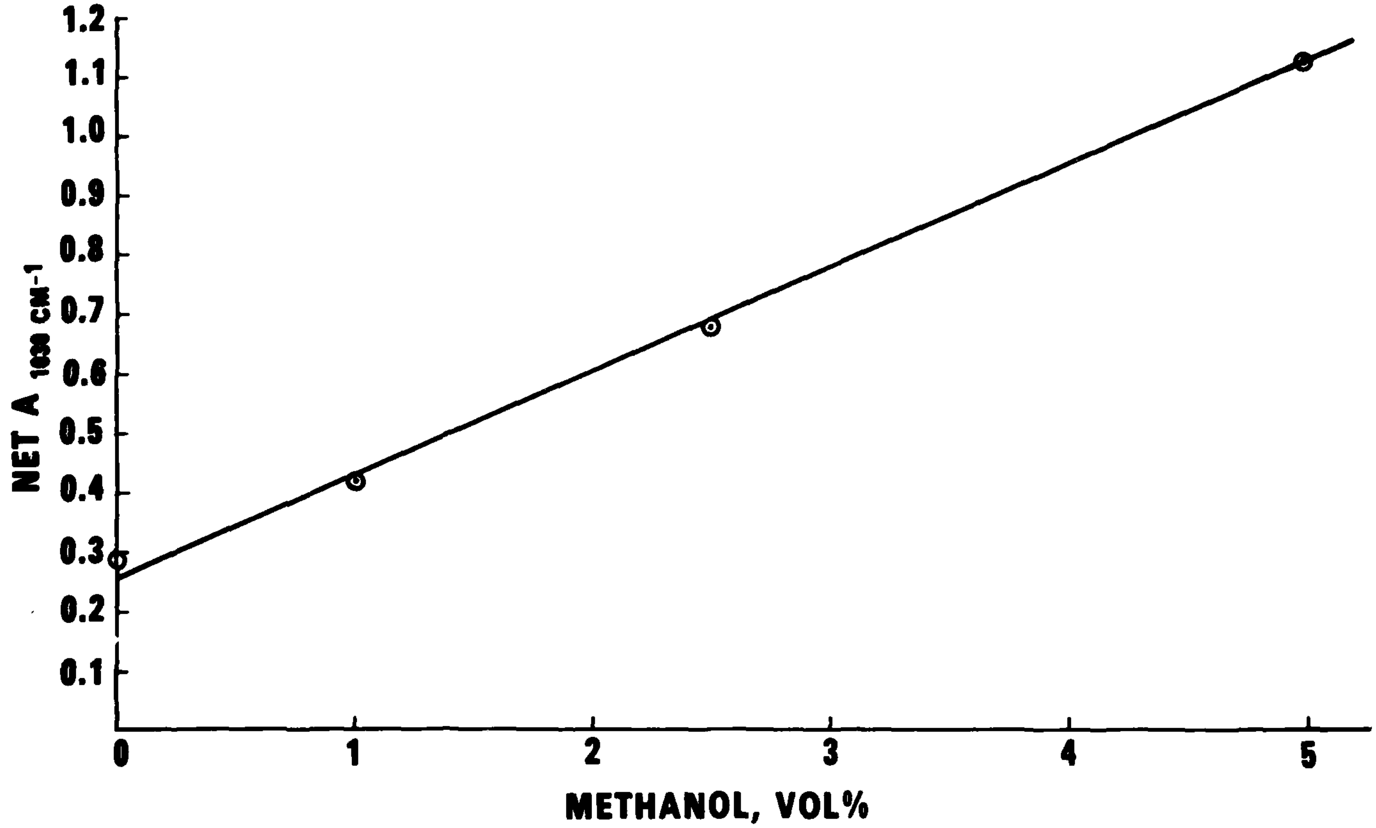

FIGURE 5. INFRARED RESPONSE FOR METHANOL IN GASOLINE

```
QUANT ROUTINES: 1-PT PROGRAM 2-NORMALIZE
3-ARRAYBUILD 4-ANALYSIS 5-ENTER ARRAY

############ POINT PROGRAM ############
ENTER  NUMBER OF POINTS
5
POINTS MUST BE ENTERED IN
DECREASING  ORDER
ENTER A WAVENUMBER
1255 CM-1
ENTER A WAVENUMBER
1230 CM-1
ENTER A WAVENUMBER
1030 CM-1
ENTER A WAVENUMBER
0967 CM-1
ENTER A WAVENUMBER
0860 CM-1
ENTER NUMBER OF READINGS TO AVERAGE
09
PUSH START
#1     1255 CM-1     77.3  %T
#2     1230 CM-1     78.6  %T
#3     1030 CM-1      8.2  %T
#4     0967 CM-1     61.3  %T
#5     0860 CM-1     78.3  %T


QUANT ROUTINES: 1-PT PROGRAM 2-NORMALIZE
3-ARRAYBUILD 4-ANALYSIS 5-ENTER ARRAY
```

FIGURE 6. POINT PROGRAM PRINTOUT

possible variability in concentration. The absorbance values obtained for the base gasoline were stored in Array Row 1 of the net normalized absorbance matrix and were used to correct the absorbance values at the other analytical frequencies.

A POINT PROGRAM and NORMALIZE MODE were run for each low and high standard of the alcohol components. The low standard, usually 1 percent alcohol, was run first, and the high standard, usually 10 percent alcohol, was then run as an intercept correction. The original concentration of the alcohol (1 percent) and the analyte band number of that particular alcohol were identified as required by the program. The absorbance values obtained for the high standard were entered in the same array row as the values for the low standard. NORMALIZE printouts for the two standards are shown in Figure 7. After all the components had been entered, the normalized absorbance matrix was inverted using the ARRAY BUILD MODE which formed the analysis matrix shown in Figure 8.

A sample was scanned using the ANALYSIS MODE of the quantitative routines which recalled the matrix set up by the computer, measured the data at the specified frequencies, and computed the concentrations of the component present. The sample was normalized to 100 percent since the sum of all components was 100 percent. Figure 9 shows an ANALYSIS printout.

C. Sample Preparation and Handling

Blends of alcohols with different base fuels were analyzed to study the gasoline matrix effect. Since different gasolines have variable compositions, a new quantitative program was established for each new gasoline to be analyzed. To identify which alcohols were present as analytes, a scan of the blend was obtained and examined for peaks at the respective analyte wavelengths. If the base fuel was not readily available, it was recovered by removing the alcohols by water extraction.

A 100-ml sample was extracted using two 50-ml portions of water. After the washings, the extracted gasoline was filtered over Na_2SO_4. The recovered fuel was then used to prepare the low and high standards needed to set up the new quantitative program.

Because methanol severely attacks the NaCl cell, methanol was run using an Irtran cell. After being placed in the infrared spectrophotometer, the cell was allowed to warm up for approximately 3-5 minutes to reach a constant pathlength before analysis.

```
************  NORMALIZE  ************
01     1255 CM-1     81.3  %T
02     1230 CM-1     82.0  %T
03     1030 CM-1     51.1  %T
04     0967 CM-1     63.9  %T
05     0860 CM-1     81.6  %T

ENTER BACKGROUND NUMBERS
01
ENTER BACKGROUND NUMBERS
02
ENTER BACKGROUND NUMBERS
05
ENTER PATHLENGTH
.020
ENTER CONCENTRATION
0001
ENTER SOLVENT CONCENTRATION
0099
ENTER ARRAY ROW NUMBER
2
IS THIS AN INTERCEPT CORRECTION?
1 FOR YES     0 FOR NO
NO
PUSH START
ROW  =  0 2
PATHLENGTH  =  .020
CONCENTRATION  =  0001
SOLVENT  CONCENTRATION = 0099
1030 CM-1          1.0005 A
0967 CM-1          0.0101 A
```

```
************  NORMALIZE  ************
01     1255 CM-1     77.3  %T
02     1230 CM-1     78.6  %T
03     1030 CM-1      0.2  %T
04     0967 CM-1     61.3  %T
05     0860 CM-1     78.3  %T

ENTER BACKGROUND NUMBERS
01
ENTER BACKGROUND NUMBERS
02
ENTER BACKGROUND NUMBERS
05
ENTER PATHLENGTH
.020
ENTER CONCENTRATION
0010
ENTER SOLVENT CONCENTRATION
0090
ENTER ARRAY ROW NUMBER
2
IS THIS AN INTERCEPT CORRECTION?
1 FOR YES     0 FOR NO
YES
IS THIS THE FIRST CORRECTION?
1 FOR YES     0 FOR NO
YES
ENTER ORIGINAL CONCENTRATION
0001
ENTER ANALYTE BAND 0
3
PUSH START
ROW  =  0 2
PATHLENGTH  =  .020
CONCENTRATION  =  0010
SOLVENT  CONCENTRATION = 0090
1030 CM-1          3.0007 A
0967 CM-1          0.0305 A
INTERCEPT = -        0.035
CORRECTED ABSORBTIVITY =          3.124
```

FIGURE 7. NORMALIZE PRINTOUTS FOR TWO STANDARDS

```
############ ARRAY BUILD ############
IS THIS AN INTERCEPT CORRECTION?
1 FOR YES     0 FOR NO
YES
ENTER ANALYSIS #
1
ENTER SLIT TYPE:
0-RESOLUTION 1-NORMAL
NORMAL SLIT
ROW  =  # 1
1030 CM-1            0.0553 A
0967 CM-1            0.0389 A
ROW  =  # 2
1030 CM-1            3.1243 A
0967 CM-1            0.0305 A
INTERCEPT ROW
1030 CM-1      -     0.0346 A
0967 CM-1            0.0000 A
MODIFY? 1-YES 0-NO
PUSH START
ROW  =  # 1
1030 CM-1      -    0.2543
0967 CM-1           0.3246
ROW  =  # 2
1030 CM-1          26.0477
0967 CM-1      -    0.4610
INVERTED INTERCEPTS
1030 CM-1      -    0.0088
0967 CM-1           0.0112

############    STANDBY    ############
```

FIGURE 8. ARRAY BUILD MODE PRINTOUT

```
############   ANALYSIS    ############
ENTER ANALYSIS #
1
ENTER NUMBER OF READINGS TO AVERAGE
09
ENTER PATHLENGTH
.045
NORMALIZE ANSWERS TO 100% ?
1 FOR YES      0 FOR NO
YES
PUSH START
ANALYSIS
NORMAL SLIT
GAIN SETTING = 040
#1      1255 CM-1      49.0   %T
#2      1230 CM-1      45.8   %T
#3      1030 CM-1       0.1   %T
#4      0967 CM-1      29.5   %T
#5      0860 CM-1      39.8   %T

1030 CM-1           2.6209 A
0967 CM-1           0.1404 A
COMPONENT 1          80.0206
COMPONENT 2          19.9794
```

FIGURE 9. ANALYSIS PRINTOUT

IV. RESULTS

Single-component standards of known concentrations were run in tests of repeatability and reproducibility. Low standard and average deviations for all alcohols are shown in Table 2. In tests of repeatability, the relative deviations remained low for the 1- and 10-vol% blends. The 20-vol% blends have higher standard deviations, but this concentration is not normally encountered. The relative deviations, which remained fairly constant for all blends, were larger for the reproducibility tests due to the decreased transmittance of the Irtran cells.

Several multicomponent mixtures of methanol, ethanol, and _t_-butanol were prepared and analyzed. Table 3 shows the results of these analyses. Compared to the single-component analysis shown in Table 2, a multicomponent analysis was not quite as accurate due to the functional group similarity of the alcohols. However, this is a good method to identify the relative concentrations of any alcohols present. A single-component analysis has been recommended to be set up for MIBK at 1725 cm^{-1}, since MIBK does not use the same background points of 1255 cm^{-1}, 1230 cm^{-1}, and 860 cm^{-1} as used for alcohols.

Actual analysis of an individual sample required only five minutes. Approximately thirty minutes is needed to set up a single-component quantitative program. An extra twenty minutes is needed for each additional alcohol, and if necessary, a water extraction can be completed in thirty minutes.

TABLE 2. ALCOHOL-GASOLINE BLENDS: IR ANALYSIS

Repeatability

Alcohol	Label	5 Runs Value, %	Stan. Dev.	Avg. Dev.
MeOH*	1%	1.04	0.01	0.01
	10%	9.49	0.09	0.07
	20%	19.39	0.14	0.12
EtOH	1%	1.18	0.02	0.01
	10%	9.73	0.02	0.02
	20%	19.45	0.04	0.03
IPA	1%	1.16	0.01	0.004
	10%	9.58	0.05	0.04
	20%	18.90	0.24	0.18
TBA	1%	1.01	0.01	0.01
	10%	9.73	0.03	0.02
	20%	19.87	0.06	0.05
MTBE	1%	1.01	0.05	0.04
	10%	10.37	0.06	0.04
	20%	17.97	0.21	0.17

Reproducibility

Alcohol	Label	5 Runs Value, %	Stan. Dev.	Avg. Dev.
MeOH*	1%	0.99	0.05	0.03
	10%	8.81	0.11	0.08
	20%	20.91	0.15	0.11
EtOH	1%	1.10	0.02	0.01
	10%	9.88	0.06	0.05
	20%	19.13	0.05	0.04
IPA	1%	1.16	0.01	0.01
	10%	9.86	0.04	0.03
	20%	18.24	0.08	0.05
TBA	1%	1.00	0.01	0.004
	10%	10.06	0.05	0.04
	20%	19.55	0.04	0.05
MTBE	1%	0.96	0.003	0.003
	10%	10.13	0.04	0.04
	20%	20.11	0.08	0.06

*Irtran cell used with 4 runs.

TABLE 3. MULTICOMPONENT ANALYSIS

Sample	Component	Known vol%	Determined vol%
A	MeOH	1	1.15
	EtOH	9	9.58
	TBA	5	5.35
B	MeOH	2.75	3.16
	EtOH	10.0	12.05
	TBA	2.75	2.95
C	MeOH	2.75	3.08
	EtOH	---	0.06
	TBA	2.75	3.01

V. CONCLUSIONS

The infrared method for oxygenates is a quick, economical way to quantitatively determine the presence of alcohols in gasoline/alcohol blends. The Beckman Microlab 620MX computing infrared spectrophotometer is easily programmed for this analysis.

Both single- and multicomponent analyses were run using several different alcohols. However, due to the functional group similarity of the alcohols, the multicomponent analysis was not as accurate. The use of Irtran cells for methanol blends increases the relative deviations due to lower transmittance. When not readily available to prepare the standards, the base fuel is recovered by water extraction of the alcohols.

Once the quantitative program is set up for a particular gasoline, an actual analysis requires only five minutes, and tests of repeatability and reproducibility have shown low standard deviations for each alcohol. Multicomponent standards are also easily set up for methanol, ethanol, and t-butanol mixtures, but a single-component program using different background points is needed for methyl-t-butyl ether.

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MICHIGAN ARMY MISSILE PLANT
PROG MGR, FIGHTING VEHICLE SYS
ATTN DRCPM-FVS-SE 1
WARREN MI 48090

PROJ MGR, M60 TANK DEVELOPMENT
ATTN DRCPM-M60-E (MR WESALA) 1
WARREN MI 48090

PROG MGR, M113/M113A1 FAMILY
OF VEHICLES
ATTN DRCPM-M113 1
WARREN MI 48090

PROJ MGR, MOBILE ELECTRIC POWER
ATTN DRCPM-MEP-TM 1
7500 BACKLICK ROAD
SPRINGFIELD VA 22150

OFC OF PROJ MGR, IMPROVED TOW
VEHICLE
US ARMY TANK-AUTOMOTIVE R&D CMD
ATTN DRCPM-ITV-T 1
WARREN MI 48090

CDR
US ARMY EUROPE & SEVENTH ARMY
ATTN AEAGC-FMD 1
APO NY 09403

PROJ MGR, PATRIOT PROJ OFC
ATTN DRCPM-MD-T-G 1
US ARMY DARCOM
REDSTONE ARSENAL AL 35809

CDR
THEATER ARMY MATERIAL MGMT
CENTER (200TH)
DIRECTORATE FOR PETROL MGMT
ATTN AEAGD-MM-PT-Q (MR PINZOLA) 1
ZWEIBRUCKEN
APO NY 09052

CDR
US ARMY RESEARCH OFC
ATTN DRXRO-EG 1
DRXRO-CB (DR GHIRARDELLI) 1
DRXRO-TT (DR SCHMIEORSHOFF) 1
P O BOX 12211
RSCH TRIANGLE PARK NC 27709

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US ARMY R&T LAB
ADVANCED SYSTEMS RSCH OFC
ATTN MR D WILSTED 1
AMES RSCH CTR
MOFFITT FIELD CA 94035

CDR
TOBYHANNA ARMY DEPOT
ATTN SDSTO-TP-S 1
TOBYHANNA PA 18466

DIR
US ARMY MATERIALS & MECHANICS
RSCH CTR
ATTN DRXMR-E 1
DRXMR-T 1
DRXMR-R 1
WATERTOWN MA 02172

CDR
US ARMY DEPOT SYSTEMS CMD
ATTN DRSDS 1
CHAMBERSBURG PA 17201

CDR
US ARMY WATERVLIET ARSENAL
ATTN SARWY-RDD 1
WATERVLIET NY 12189

CDR
US ARMY LEA
ATTN DALO-LEP 1
NEW CUMBERLAND ARMY DEPOT
NEW CUMBERLAND PA 17070

CDR
US ARMY GENERAL MATERIAL &
PETROLEUM ACTIVITY
ATTN STSGP-PW (MR PRICE) 1
SHARPE ARMY DEPOT
LATHROP CA 95330

CDR
US ARMY FOREIGN SCIENCE & TECH
CENTER
ATTN DRXST-MT1 1
FEDERAL BLDG
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CDR
DARCOM MATERIAL READINESS
SUPPORT ACTIVITY (MRSA)
ATTN DRXMD-MD 1
LEXINGTON KY 40511

HQ, US ARMY T&E COMMAND
ATTN DRSTE-TO-O 1
ABERDEEN PROVING GROUND, MD 21005

HQ, US ARMY ARMAMENT R&D CMD
ATTN DRDAR-SCM-OO (MR MUFFLEY) 1
DRDAR-TST-S 1
DOVER NJ 07801

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AVIATION MATERIAL READINESS
COMMAND
ATTN DRSTS-MEG (2) 1
DRCPO-PDE (LTC FOSTER) 1
4300 GOODFELLOW BLVD
ST LOUIS MO 63120

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P O BOX 4005
CHAMPAIGN IL 61820

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ATTN ATCD-SL (MR RAFFERTY) 1
ATCD-TA 1
ATCD-D 1
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PROPULSION LABORATORY
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CDR
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ATTN DRDNA-YEP (DR KAPLAN) 1
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US ARMY QUARTERMASTER SCHOOL
ATTN ATSM-CTD-MS 1
ATSM-TNG-PT (COL VOLPE) 1
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PENSACOLA FL 32508

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HQ, US MARINE CORPS
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CP6, RM 606 MAT-08E (MR ZIEM) 1
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ALBANY GA 31704

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